Indian J. Phys. **48**, 712–714 (1974)

Effect of annealing on the etching of the radiation damages in micas

M. K. NAGPAL AND K. K. NAGPAUL

Physics Department, Kurukshetra University, Kurukshetra 132119

(*Received* 21 *September* 1973, *revised* 18 *December* 1973)

The annealing experiments on micas are performed to learn the behaviour of etching phenomenon with the thermally annealed radiation damages. It has been observed that the healed up damages can be recovered by overetching and the probability for revealing the healed up damages with overetching increases with the amount of annealing suffered by the samples.

1. INTRODUCTION

Optical detection technique (Fleischer *et al* 1965a) of radiation damages in solids require that the damaged trails be penetrated by an etchant and therefore phenomenon of etching under various experimental conditions poses an interesting problem of study. The effect of temperature, concentration and duration of time on the etching have been studied by many workers (Blanford *et al* 1970a, 1970b) and a lot of investigations have also been made to study track annealing behaviour in various solids (Fleischer *et al* 1964, 1965b, Mehta & Rama 1969, Nagpal *et al* 1974). But no attention has been paid so far to the combined effects of etching and annealing processes of tracks formation.

The present work was undertaken to study what effect annealing would have on the etching of the radiation damages in micas.

2. EXPERIMENT AND RESULTS

Large, thick, single crystal samples of the Rajasthan micas having uniform U-concentration ($\sim 5\times10^{-11}$ atom/atom in case of muscovite and $\sim 0{\cdot}6\times10^{-9}$ atom/atom in case of biotite) were irradiated in the thermal column of CIRUS atomic reactor at B.A.R.C., Bombay with a dose of $\sim 10^{17}$ nvt and $\sim 10^{16}$ nvt for muscovite and biotite respectively, to produce a known density of induced tracks in the samples. The tracks thus produced are only fission tracks. A large number of pieces were cut from each sample and annealed for one hour at temperature ranging from 400°C to 600°C for muscovite and from 300°C to 400°C for biotite. Each piece was then cleaved to expose a fresh surface* and etched twice in 40% HF at 27°C for two distinct times; firstly normal etching—the time duration for this etching was kept ~5 seconds (figure 1) for biotite and ~30 minutes (figure 2) for muscovite and secondly the same samples were over-etched, the duration for this etching was kept ~1 minute (figure 3) for biotite and ~3 hours (figure 4) for muscovite. The track densities in each case were measured in annealed samples. The ratio $\rho(0)/\rho(N)$ of the track densities of overetched [$\rho(0)$] and normal etched [$\rho(N)$] tracks for different annealing temperature computed and plotted against the temperature as shown in figure 5.

3. Discussion

In the earlier experiments (Fleischer *et al* 1964, Mehta & Rama 1969) it has been shown that

1. The etchable range and track density decreases gradually with increase in annealing temperature.

2. Tracks appear to shorten before their number per unit area decrease.

This experiment besides confirming the above observations gives us an additional information (figure 5) regarding the probability of revealing the healed up damages due to thermal affection by overetching and shows that it increases with the severity of the annealing. In general, prolonged etching can give rise to increase in track density due to the following factors

1. It can reveal additional tracks as the surfaces are progressively dissolved and new surfaces which come up start contributing to the tracks number per unit area. In the present study the contribution of this factor for the net increase of the tracks can be neglected as the bulk etching rate of micas is very small; no doubt this factor can be very prominent one in the materials in which the bulk etching rate is large such as in glasses etc. Combining the contribution of this factor with annealing which decreases the track density, the variation of the ratio of $\rho(0)/\rho(N)$ should either be almost a horizontal line or a decreasing curve depending on their relative contributions. Therefore, this factor do not account for the observed nature of the curves.

2. It can force the partially healed up tracks to be etched. It is possible because even with complete annealing in strict sense, one can not claim, the recovery of the damaged region as exactly the original healthy material due to universal law of entropy.

* For annealing study the need of the fresh tracks is found to be essential as it is observed that surface tracks are almost unaffected as far as annealing is concerned, the reason is not yet known but might be due to some surface effects.

The amount of chemical entering the damages which are responsible for etching mainly depends on the available diameter of radiation damages. For each initial diameter size—a minimum etching time is needed for the etching of the damages to be visible in optical microscope. This minimum etching time is obviously larger for smaller diameter damages. Therefore with the annealing the number of damages with smaller diameter increases and hence the probability for recovering these healed up damages after over-etching increases as is evident in figure 5.

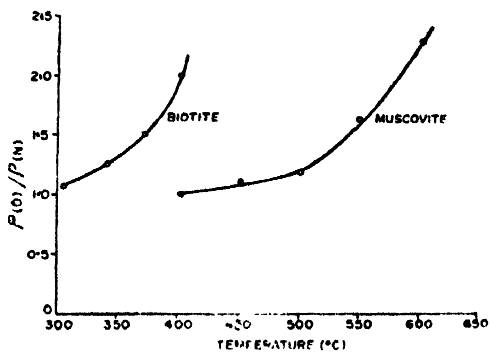

Figure 5. Plot of $\rho(0)/\rho(N)$ versus one hour annealing temperature for muscovite and biotite.

4. Conclusions

(1) The overetching can force the partially healed up damaged trails in micas to be etched as visible track under an optical microscope.

(2) The probability for revealing the tracks after overetching increases with the severity of annealing suffered by micas.

Acknowledgment

The authors would like to thank Dr. Kamal Nandi for critically reviewing the manuscript. The financial assistance from C.S.I.R. and Department of Atomic Energy are gratefully acknowledged.

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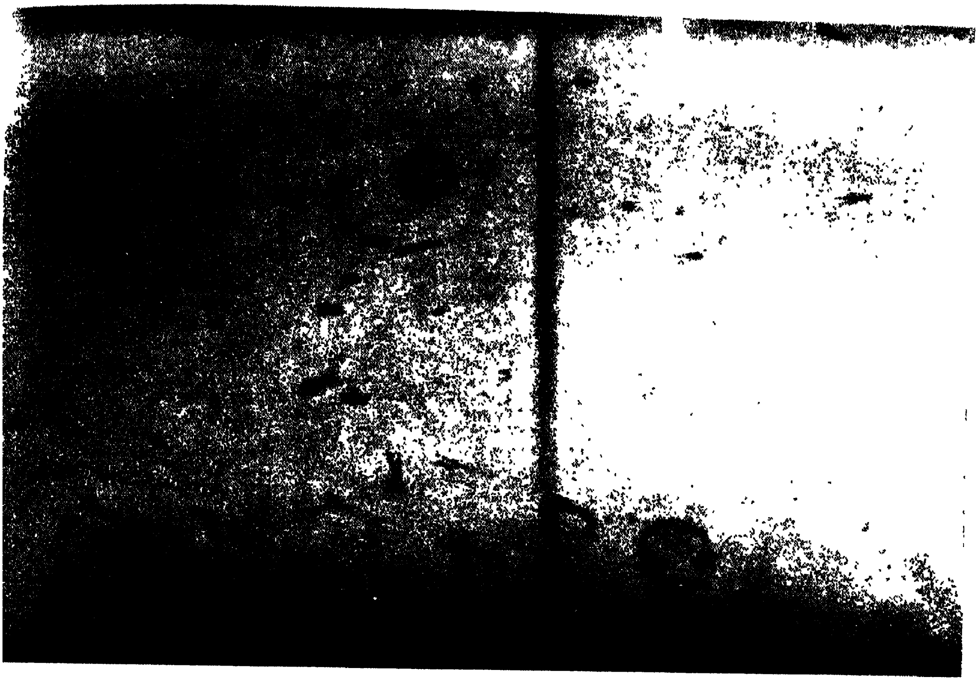

Figure 1. A photo micrograph of normal etched tracks in biotite.

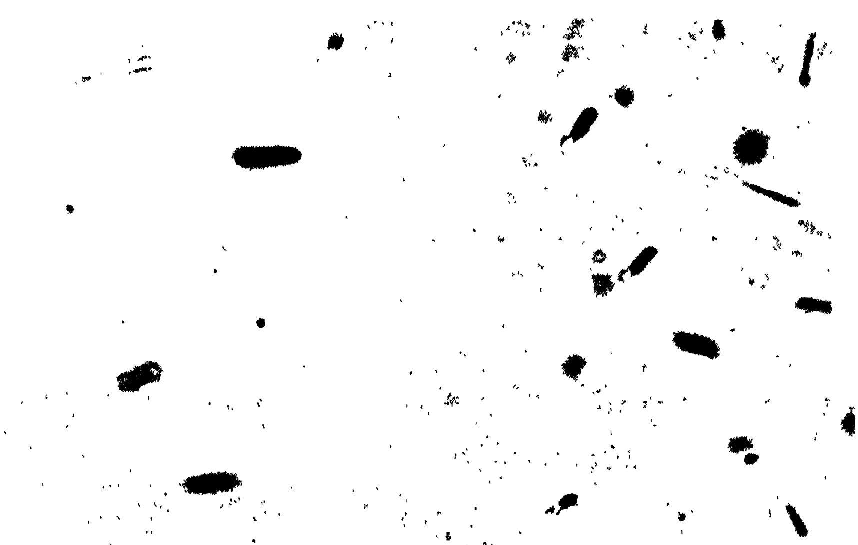

Figure 2. A photo micrograph of normal etched tracks in muscovite.

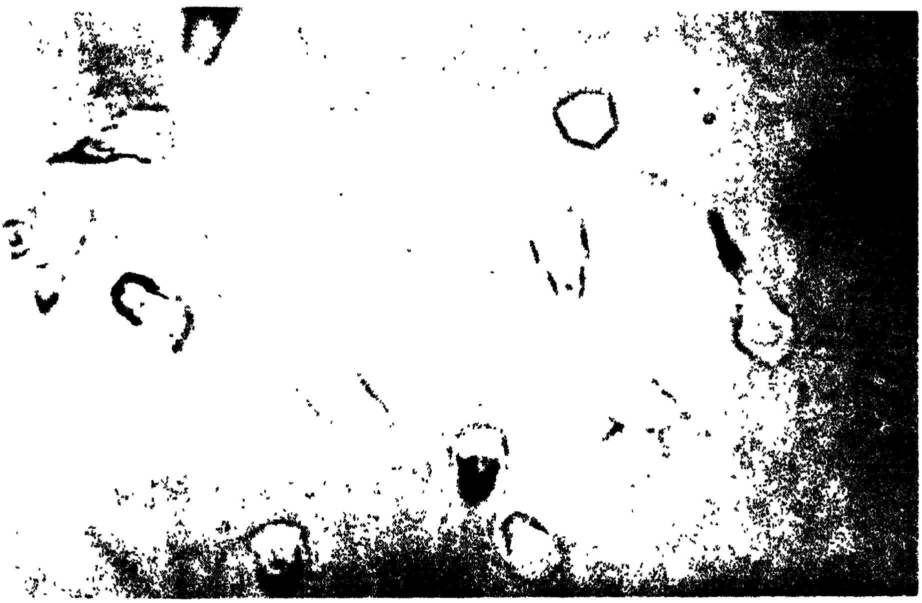

Figure 3. A photo micrograph of over-etched tracks in biotite

Figure 4. A photo micrograph of over-etched tracks in muscovite.